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REPORT OF INVESTIGATIONS — NO. 132

CHEMICAL CHARACTERISTICS OF BANDED INGREDIENTS OF COAL

By

O. W. Rees, W. F. Wagner, and W. G. Tilbury

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CHEMICAL CHARACTERISTICS OF BANDED INGREDIENTS OF COAL

BY

O. W. Rees, W. F. Wagner, and W. G. Tilbury

Abstract

Data are presented on certain fundamental chemical characteristics for four sets of banded ingredients and whole coals from Illinois representing three ranks—high volatile bituminous A, B, and C. Evidence is shown of variation by rank of the banded ingredients, vitrain, clarain, and durain. Evidence of variations by rank of fusains is more erratic. Comparison of characteristics of Hsiao fusains with those of hand-picked fusains indicates that the hand-picked samples contain rather large proportions of more reactive materials. Variations in reported analyses of banded ingredients appear to be due partially to the influence of varying rank, but also to variations in the selection of samples.

IN THE work on Illinois coals in this laboratory, considerable attention has been given to studies of the physical and chemical characteristics of the banded ingredients, vitrain, clarain, durain, and fusain. The Stopes classification (*15*) of banded ingredients is based on macrovisual appearance. It is of interest, therefore, to compare these visually selected ingredients to learn whether each shows more or less specific chemical characteristics. It is also of interest to learn whether each ingredient, secured from coals of different ranks, varies in chemical characteristics as do the source whole coals.

Published analyses for the banded ingredients show wide differences in chemical composition for each ingredient. Probably the composition of each ingredient, at least of vitrain, clarain, and durain, is influenced by the degree of metamorphosis undergone; but how far this may account for reported differences and how much may be due to variation in the selection of ingredients, source plant materials, etc., is a question. Recently Lowry (*6*), in summarizing chemical information on the banded ingredients, showed clearly the wide differences in reported analyses for each ingredient. Marshall (*7*) attempted to correlate analyses of anthraxylon with type and rank variation in coal seams. Fisher *et al.* (*3*) studied hydrogenation characteristics of the ingredients. Sprunk (*13*) summarized the influence of physical constitution of coal upon its chemical properties. Many other investigators have contributed information on various phases of this problem, but space does not permit a complete summary.

This report presents the results of studies of certain chemical characteristics on four sets of banded ingredients from the three ranks of coal—high volatile bituminous A, B, and C—represented in Illinois. The work represents an effort to obtain information on variations of chemical characteristics of ingredients as related to rank of source coals, and in addition to secure information on differences due to variability in visual selection, particularly of the fusains.

Tests on Coal Samples

Four sets of samples, including whole coals and banded ingredients from Gallatin, Franklin, Macoupin, and Henry counties were studied. These represent coals of high volatile bituminous ranks A, B, C, and C, respectively. The whole coals were channel samples, cut down, crushed to pass a 4-mesh sieve, and sealed in sample cans in the mines. The banded ingredient samples were hand-picked in the mines, the selection being made macroscopically. These samples were crushed to pass a 4-mesh sieve and were sealed in sample cans in the mines. In the laboratory, samples were air-dried, crushed to –20 mesh size in a Braun type 6CP pulverizer, and further pulverized to smaller sizes in a ball mill.

Several tests were used to compare chemical characteristics of the banded ingredients. Proximate analysis, calorific value, and total sulfur were determined on −60 mesh samples according to standard A.S.T.M. procedures (*1*). Reactivity index was determined by the C.R.L. reactivity test (*11, 12*). Determinations were made on −40 +60 mesh samples in both oxygen and air, and results were indicated as T_{15} and T_{75}, respectively. Free swelling index tests were made on −60 mesh samples according to a tentative A.S.T.M. method (*2*). Moisture characteristics were determined by the equilibration method described by Stansfield and Gilbart (*14*) and later used in this laboratory (*10*).

Further studies were made on the hand-picked fusains. Fusain determinations were made on these samples by the Hsiao method (*4*). The inert portions (Hsiao fusains) remaining after nitric acid oxidation of the hand-picked fusains were studied for reactivities by the C. R. L. method, and moisture characteristics were studied by the equilibration method. Proximate and ultimate analyses were made according to A.S.T.M. standard methods.

Analyses

Proximate analyses, total sulfur, and calorific values for whole coals and hand-picked ingredients are summarized in table 1. In general, analytical values for the vitrains, clarains, and durains vary in the same way as do those for the corresponding whole coals. Moisture and calorific values are of particular interest in considering variations due to rank. As-received moisture values for vitrain, clarain, and durain increase with decrease in rank, as do those for the whole coals. Moisture values for the fusains show an exception in regular increase with decrease of rank of the corresponding source coals; that for the Franklin County fusain is higher than that of the fusain from the lower rank Macoupin County coal. Fusain moisture values are distinctly higher than those for other ingredients.

Calorific values on the moist mineral-matter-free basis, for all ingredients, decrease with decrease in rank of the source whole coals. In general, calorific values on the dry mineral-matter-free basis for vitrain, clarain, and durain show this same trend, although values for vitrain and clarain from Henry County are higher than those for the same ingredients from Macoupin County. This is also true for the corresponding whole coals. Fusain from the Franklin County rank B coal showed the highest calorific value on the dry mineral-matter-free basis. With the exception of the fusain from the Gallatin County rank A coal, calorific values (dry mineral-matter-free) for fusains are higher than for other banded ingredients and whole coals. This is in accord with the findings of Parr, Hopkins, and Mitchell (*8*) in a study of Illinois fusains. However, the work reported by these authors covered fusains from high volatile bituminous B and C coals but not for fusain from high volatile bituminous A.

Reactivity and Free Swelling Indices

Table 1 gives reactivity indices for the samples studied. The T_{15} and T_{75} values for the whole coals decrease with decrease in rank. This same trend is exhibited also by the banded ingredients. Reactivity indices for ingredients and whole coals from the same source are similar. Results obtained by the C.R.L. test are probably more nearly a measure of the reactivity of the most reactive portion of mixed samples. Sherman *et al.* (*12*) gave evidence of this. Macroscopically picked ingredients may be mixtures containing portions of various other ingredients as impurities. This may account for the similarity of the values for samples from the same source. The fact that ingredient reactivity indices decrease with decrease in rank of corresponding whole coals is further evidence of variation of ingredients with rank.

Table 1 also gives free swelling indices for whole coals and banded ingredients. These values do not show progressive decrease with rank as do the reactivity indices. Samples from the highest rank coal from Gallatin County show the highest free swelling indices. Samples from the next

TABLE 1.—ANALYSES OF HAND-PICKED SAMPLES

	Whole coal	Vitrain	Clarain	Durain	Fusain
Moisture (as-received), %					
Gallatin	5.1	4.4	3.6	....	17.8
Franklin	8.0	9.2	8.0	6.0	21.1
Macoupin	14.0	15.3	13.4	....	20.3
Henry	19.3	17.2	18.2	16.2	25.2
Ash (dry), %					
Gallatin	10.0	8.1	10.3	....	18.9
Franklin	10.3	4.3	4.9	11.4	8.6
Macoupin	11.9	3.4	4.3	....	10.0
Henry	11.0	7.5	11.0	7.5	14.1
Volatile matter (dry, ash-free), %					
Gallatin	43.1	42.8	41.1	....	28.6
Franklin	40.6	34.1	37.3	43.2	15.2
Macoupin	45.0	43.3	47.0	....	19.6
Henry	44.0	41.6	44.9	43.6	26.9
Fixed C (dry, ash-free), %					
Gallatin	56.9	57.2	58.9	....	71.4
Franklin	59.4	65.9	62.7	56.8	84.8
Macoupin	55.0	56.7	53.0	....	80.4
Henry	56.0	58.4	55.1	56.4	73.1
Total S (dry), %					
Gallatin	3.19	2.42	2.74	....	4.26
Franklin	1.69	1.04	1.37	1.16	4.20
Macoupin	5.23	3.32	4.16	....	6.20
Henry	4.77	4.76	4.32	3.85	10.46
Calorific value, B.t.u.					
Moist mineral-matter-free					
Gallatin	14,098	14,371	14,361	.....	11,719
Franklin	13,377	13,170	13,386	13,827	11,634
Macoupin	12,022	11,920	12,248	.....	11,428
Henry	11,486	11,667	11,633	12,131	10,397
Dry mineral-matter-free					
Gallatin	14,989	15,108	14,975	.....	14,985
Franklin	14,701	14,576	14,633	14,849	15,155
Macoupin	14,349	14,194	14,284	.....	14,812
Henry	14,694	14,383	14,644	14,739	14,847
Free swelling index					
Gallatin	7.0	6.5	6.5	...	1+
Franklin	3.0	4.0	3.0	1.0	1.0
Macoupin	4.5	3.5	3.5	...	2.0
Henry	4.5	...	3.5	3.5	1+
Reactivity indices, °C.					
T_{15} (in oxygen)					
Gallatin	207	210	211	...	211
Franklin	182	184	187	180	186
Macoupin	160	160	159	...	179
Henry	164	...	163	175	170
T_{75} (in air)					
Gallatin	254	258	254	...	258
Franklin	229	245	240	218	228
Macoupin	188	190	190	...	222
Henry	186	...	185	193	208

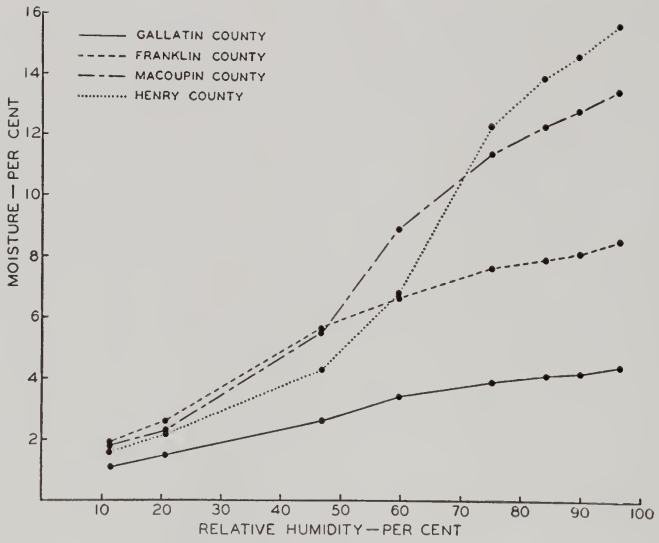

Fig. 1.—Moisture characteristics of whole coals

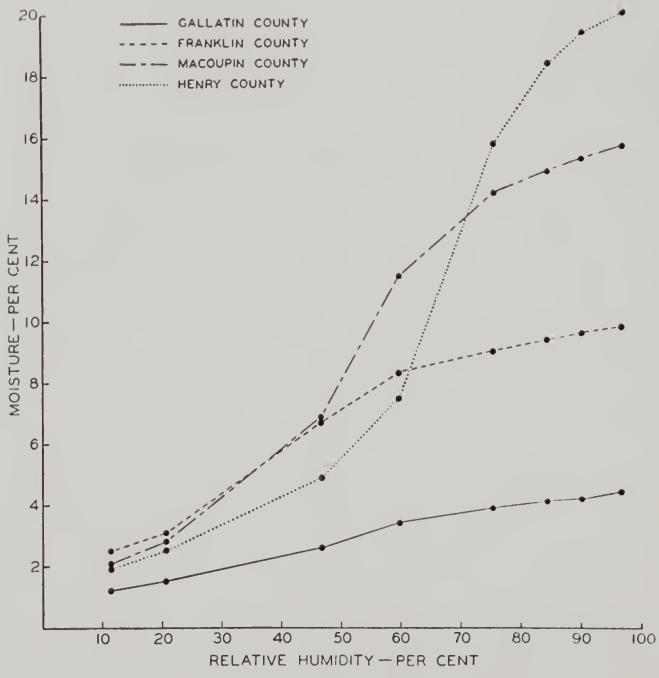

Fig. 2.—Moisture characteristics of vitrains

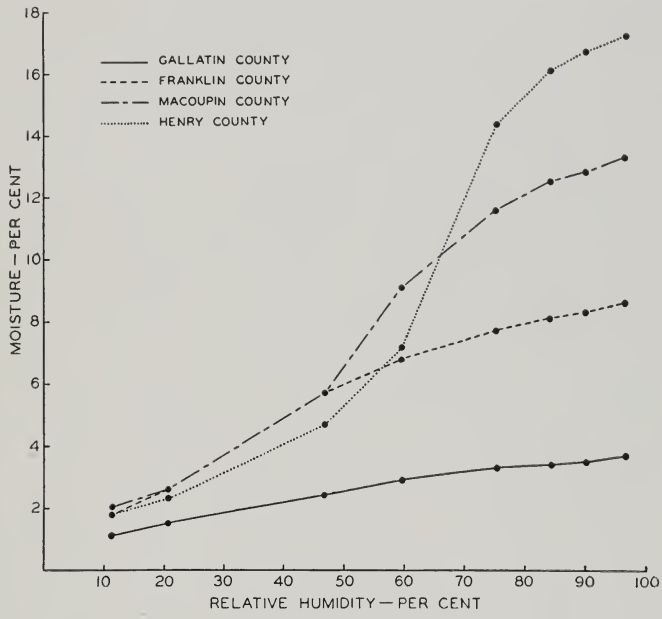

Fig. 3.—Moisture characteristics of clarains

highest rank coal from Franklin County show the lowest. Values for the two C rank coals appear to be similar for both whole coals and banded ingredients. Fusain is non-swelling, and values greater than 1 shown in table 1 indicate the presence of small amounts of swelling ingredients in the hand-picked fusains.

Moisture Characteristics

Figures 1 to 5 show graphically the results obtained. Samples, ground to pass a 14-mesh sieve, were brought to equilibrium at different humidities, and moisture contents were then determined by heating in a vacuum oven at 105° C. for 3 hours. These

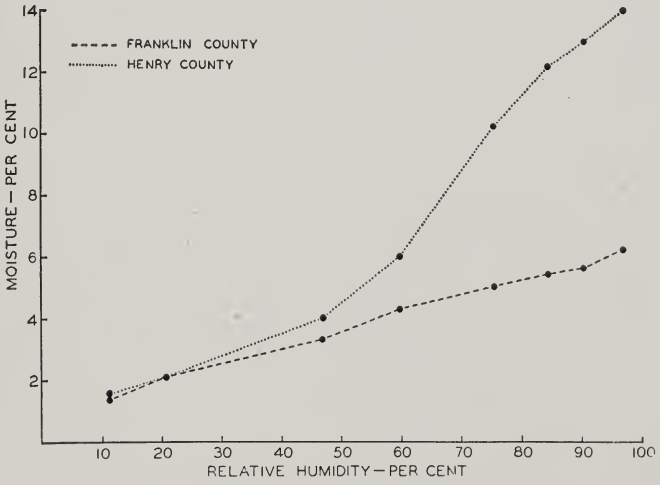

Fig. 4.—Moisture characteristics of durains

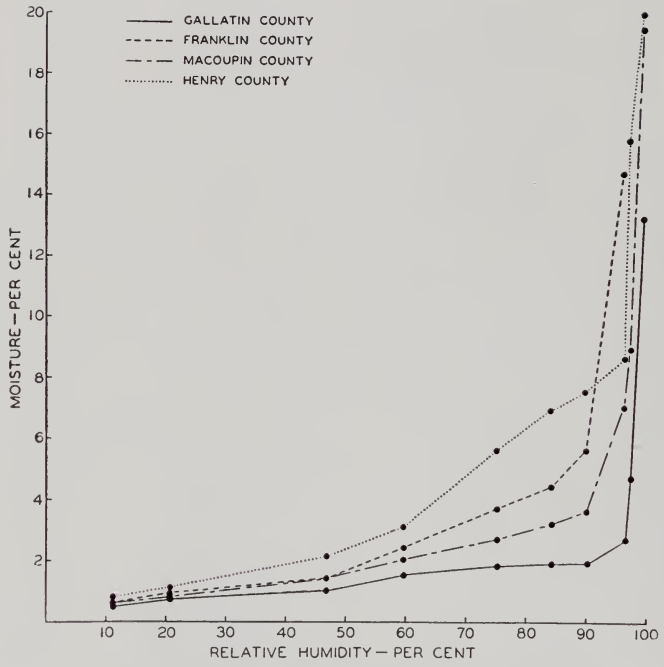

Fig. 5.—Moisture characteristics of hand-picked fusains

values were plotted against the corresponding relative humidities. For the four sets of samples studied, the interrelations of moisture-humidity curves are similar. In general, vitrain curves are highest, clarain and whole coal are next, durain is somewhat lower, and fusain is decidedly lower up to high humidities where the curves rise steeply. Comparisons of the moisture-humidity curves for the four whole coals studied, as well as for the corresponding banded ingredients, show wide differences. Curves for vitrains, clarains, and durains have the same general shape and occupy the same relative positions as do curves for the different rank whole coals. The fusain curves (fig. 5) do not occupy the same relative positions.

These comparisons appear to show that moisture characteristics indicate variations by rank in vitrain, clarain, and durain as in the whole coals. Indications of variation by rank of fusains are not clear.

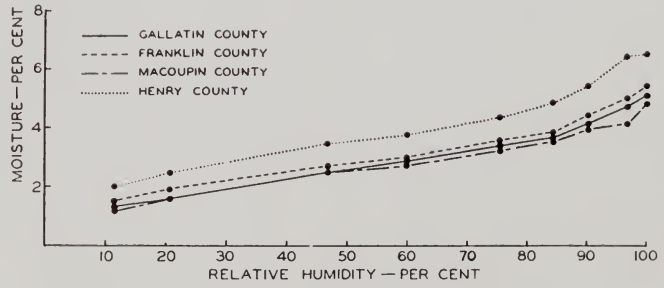

Fig. 6.—Moisture characteristics of Hsiao fusains

Further Studies on Fusains

The question of purity of banded ingredient samples made it desirable to study the characteristics of more carefully purified samples. The relative inertness of fusain to nitric acid (*4*) makes possible its purification, whereas no such means of purification of the other ingredients is available. Therefore further studies were made on the fusains in an effort to learn whether distinct differences in chemical characteristics of the hand-picked fusains were due to the presence of various amounts of other ingredients or to the influence of rank variation of the source coals.

Determinations were made by the Hsiao method (*4*) on the hand-picked fusains. Furthermore, since fusain is quite friable, similar tests were made on the close-sized reactivity samples (−40 +60 mesh) to determine the loss of fusain in sizing between close limits:

County	% Hsiao Fusain in	
	Hand-picked fusains	−40+60 mesh fusain
Gallatin	61.5	31.6
Franklin	79.3	46.8
Macoupin	65.0	44.1
Henry	49.0	28.2

This tabulation shows that the hand-picked samples fall far short of being 100% fusain, as judged by the Hsiao fusain determinations. Close sizing further reduces the fusain content of −40 +60 mesh samples. The question then arose as to whether results obtained on samples selected according to the Stopes classification by visual appearance are to be interpreted as representative of fusain or of mixtures in which fusain may in some cases be the lesser constituent as judged by chemical characteristics.

Hsiao Fusains

Since the Hsiao determinations on the four hand-picked fusains showed the presence of considerable amounts of oxidizable material, it was thought that residues from nitric acid treatment might be more similar in characteristics. Accordingly, such inert portions were prepared from the four samples of fusain available. Five 10–12 gram portions of each sample were refluxed in 200–240 ml. of 8 *N* nitric acid for 7 hours. The inert residues were purified by removing alkali-soluble material with 1 *N* sodium hydroxide, followed by repeated centrifuging and final washing with dilute hydrochloric acid and water. The samples were then dried by exposure to the laboratory atmosphere (with frequent stirring) for 8 or 9 hours, and then various determinations were made. The nitric acid inert residues prepared as outlined are referred to here as Hsiao fusains.

Analyses.—Table 2 presents the results of proximate, ultimate, total sulfur, and calorific value analyses. Samples available were too small to permit duplicate determinations in most cases. These analyses show much closer agreement than did those for the hand-picked fusains. However, certain differences are apparent even in these samples. The ash for the Gallatin County sample is higher than for the others. The carbon value for the Henry County sample was distinctly lower than those for the other three. Nitrogen values are similar. Sulfur values for three of the samples are not very different; that for the sample from the low sulfur Franklin County coal is definitely lower than for the others.

Dry ash-free volatile matter and fixed carbon values and dry mineral-matter-free (unit coal) calorific values for the samples from Gallatin, Franklin, and Macoupin counties are very similar. The sample from Henry County shows higher volatile matter, lower fixed carbon, and lower calorific values than the other three.

To summarize, it is evident that the variations of analytical results for the hand-picked fusains were due, in large part, to the presence of other more reactive materials in the samples. Although the Hsaio fusains show comparatively small variations in certain characteristics, these variations do not appear to correlate with the ranks of the corresponding coals.

Reactivity index and specific surface.—Table 2 shows that reactivity indices for these samples are decidedly higher than for the corresponding hand-picked samples.

Table 2.—Analyses of Hsiao Fusains

County	Hsiao Fusains	Hand-picked Fusains	Hsiao Fusains
	Ash (Dry), %		C (Dry, Ash-Free), %
Gallatin	5.1	18.9	89.64
Franklin	2.3	8.6	89.56
Macoupin	3.8	10.0	89.13
Henry	2.4	14.1	87.62
	Volatile Matter (Dry, Ash-Free), %		H (Dry, Ash-Free), %
Gallatin	13.6	28.6	2.47
Franklin	13.5	15.2	2.44
Macoupin	13.8	19.6	2.57
Henry	14.8	26.9	2.43
	Fixed C (Dry, Ash-Free), %		N (Dry, Ash-Free), %
Gallatin	86.4	71.4	0.68
Franklin	86.5	84.8	0.66
Macoupin	86.2	80.4	0.55
Henry	85.2	73.1	0.87
	Total S (Dry), %		O (Dry, Ash-Free), %
Gallatin	0.42	4.26	6.77
Franklin	0.21	4.20	7.13
Macoupin	0.52	6.20	7.21
Henry	0.46	10.46	8.61
	B.t.u. (Dry Mineral-Matter-Free)		Sp. Surface Sq. Cm./Gram
Gallatin	14,103	14,985	5490
Franklin	14,076	15,155	4610
Macoupin	14,145	14,812	4170
Henry	13,969	14,847	5310
	T_{15}, ° C.		
Gallatin	285	211	
Franklin	263	186	
Macoupin	243	179	
Henry	266	170	
	T_{75}, ° C.		
Gallatin	326	258	
Franklin	310	228	
Macoupin	319	222	
Henry	313	208	

Earlier in this report attention was called to the fact that reactivity indices for ingredients and whole coals from the same source are quite similar. This is true even for the fusains. Since fusain is considered the most inert of the ingredients, its reactivity index would be expected to be higher. It was suggested that, since the C.R.L. method probably measures more nearly the reactivity of the most active constituent of a mixture, the values found were representative of the most reactive constituent in mixed samples and not of the particular banded ingredient supposedly being tested. If this were true in the case of the fusains, removal of the more reactive material should have given residues whose reactivity indices were definitely higher. This is exactly what happened in the Hsiao fusains.

Some differences in reactivity indices for the four samples are apparent. T_{15} and T_{75} values are highest for the sample from the highest rank coal. The T_{15} value is lowest for the sample from Macoupin County, and the T_{75} value lowest for the Franklin County sample. No definite correlation with rank is apparent.

It was thought that differences in reactivity indices might be due to differences in specific surface of the samples. Sherman *et al.* (*12*) showed that increasing the specific surface results in lowering reactivity index when studying the same coal. Accordingly, specific surface determinations were made by the Lea and Nurse method (*5*). Table 2 shows that the sample from Gallatin County with the highest T_{15} and T_{75} values had the highest specific surface; the sample from Macoupin County with the lowest T_{15} value showed the lowest specific surface. Although a study of the relation of surface to reactivity should be made on the same coal, the authors wished to determine whether there might be such a correlation for these samples. As the relation here is the reverse of that to be expected, it seems reasonable to assume that differences in reactivity index for these samples are due to more fundamental causes.

Moisture characteristics.—Results obtained by the equilibration method are shown graphically in figure 6. Comparison of figures 5 and 6 indicates that moisture-humidity curves for the Hsiao fusains are more similar than are the curves for the hand-picked fusains. All samples (fig. 6) show a gradually increasing moisture content with increasing humidity, but there is practically no indication of high moisture take-up at high humidities such as that exhibited by the hand-picked fusains.

At present there is no definite explanation for this difference between the Hsiao and hand-picked fusains. Previous data (*9*) indicate that the differences in moisture characteristics cannot be due to the presence or absence of water-soluble salts. It seems reasonable to suppose that possible changes in porosity or surface characteristics, or both, resulting from nitric acid oxidation might account for the differences in moisture characteristics between hand-picked and Hsiao fusains.

In an attempt to secure further information regarding banded ingredients of Illinois coals, some work has been done with the electron microscope. Through the cooperation of G. L. Clark, of the University of Illinois, electron microscope pictures at various magnifications were made for one set of banded ingredients and for the four Hsiao fusains. This work appears promising but has not progressed far enough to permit definite conclusions to be drawn.

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